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(54) PRODUCTION DE PEAUX A PARTIR DE DISPERSIONS DE  
 (54) PRODUCTION OF SKINS FROM POLYURETHANE DISPERSIONS

(57) A process for producing skins from polyurethane dispersions comprises I. producing on a mold a sheetlike layer of an aqueous dispersion comprising a polyurethane (A), the mold surface being free of any electrolyte which would cause the aqueous dispersion comprising a polyurethane (A) to coagulate, II. drying the film, produced in step (I), of the aqueous dispersion comprising a polyurethane (A) to obtain a single-layered skin, III. optionally producing a skin composed of a plurality of skin layers by producing on the mold, obtained in step II, coated with a single-layered skin a further sheetlike layer of an aqueous dispersion comprising a polyurethane (A), IV. drying the new film, formed in step (III), of the aqueous dispersion comprising a polyurethane (A) to form a further skin layer, and V. optionally repeating steps (III) and (IV) from 5 to 50 times.



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(54) **PRODUCTION DE PEAUX A PARTIR DE DISPERSIONS DE**  
(54) **PRODUCTION OF SKINS FROM POLYURETHANE**  
**DISPERSIONS**

(57) A process for producing skins from polyurethane dispersions comprises I. producing on a mold a sheetlike layer of an aqueous dispersion comprising a polyurethane (A), the mold surface being free of any electrolyte which would cause the aqueous dispersion comprising a polyurethane (A) to coagulate, II. drying the film, produced in step (I), of the aqueous dispersion comprising a polyurethane (A) to obtain a single-layered skin, III. optionally producing a skin composed of a plurality of skin layers by producing on the mold, obtained in step II, coated with a single-layered skin a further sheetlike layer of an aqueous dispersion comprising a polyurethane (A), IV. drying the new film, formed in step (III), of the aqueous dispersion comprising a polyurethane (A) to form a further skin layer, and V. optionally repeating steps (III) and (IV) from 5 to 50 times.



## Production of skins from polyurethane dispersions

### Abstract

A process for producing skins from polyurethane dispersions comprises

- I. producing on a mold a sheetlike layer of an aqueous dispersion comprising a polyurethane (A), the mold surface being free of any electrolyte which would cause the aqueous dispersion comprising a polyurethane (A) to coagulate,
- II. drying the film, produced in step (I), of the aqueous dispersion comprising a polyurethane (A) to obtain a single-layered skin,
- III. optionally producing a skin composed of a plurality of skin layers by producing on the mold, obtained in step II, coated with a single-layered skin a further sheetlike layer of an aqueous dispersion comprising a polyurethane (A),
- IV. drying the new film, formed in step (III), of the aqueous dispersion comprising a polyurethane (A) to form a further skin layer, and
- V. optionally repeating steps (III) and (IV) from 5 to 50 times.

Production of skins from polyurethane dispersions

5 Description

The present invention relates to processes for producing skins from polyurethane dispersions, which comprise

- 10 I. producing on a mold a sheetlike layer of an aqueous dispersion comprising a polyurethane (A), the mold surface being free of any electrolyte which would cause the aqueous dispersion comprising a polyurethane (A) to coagulate,
- 15 II. drying the film, produced in step (I), of the aqueous dispersion comprising a polyurethane (A) to obtain a single-layered skin,
- 20 III. optionally producing a skin composed of a plurality of skin layers by producing on the mold, obtained in step II, coated with a single-layered skin a further sheetlike layer of an aqueous dispersion comprising a polyurethane (A),
- 25 IV. drying the new film, formed in step (III), of the aqueous dispersion comprising a polyurethane (A) to form a further skin layer, and
- 30 V. optionally repeating steps (III) and (IV) from 5 to 50 times.

Skins from polyurethane dispersion are known from JP-A S63-256409, for example. The skins are produced therein by dipping a salt wetted mold into a polyurethane dispersion and subsequently subjecting the film formed to thermal curing. The  
35 skins produced in this way can be backfoamed and be used for example for the interior of automobiles. The disadvantage of this process is that the mold has to be coated with a salt prior to every processing step. This process is relatively complicated, since the mold has to be coated with a salt in a separate  
40 operating step.

DE-A-19708451, unpublished at the priority date of the present invention, discloses the production of thin-walled elastic  
45 articles by producing on the surface of a mold a layer of an electrolyte, dipping the mold into a polyurethane dispersion, coagulating the polyurethane dispersion on the surface, optionally washing and curing the resulting layer of coagulated

polyurethane particles, and removing the articles formed from the mold.

5 EP-A-0257225 discloses heat coagulating heat sensitive  
polyurethane dispersions to produce hollow articles. These  
dispersions consist for example of a polyurethane rendered  
hydrophilic by ionic groups, water-soluble polyether  
polyurethanes as described for example in DE-A 2516979, 2534304  
and 3330197 and an electrolyte, for example a metal salt.

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It is an object of the present invention to provide a process for  
producing skins from aqueous dispersions comprising a  
polyurethane without the disadvantages of prior art processes.

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We have found that this object is achieved by the process  
described at the beginning.

20 The molds on which the sheetlike layer of an aqueous dispersion  
of polyurethane (A) is produced in step (I) are molds of the type  
generally customary for producing automotive interior parts such  
as dashboards, door linings and column linings. Such molds are  
described in WO 93/23237 and JP-A-S63-256409, for example. The  
molds preferably have surface structures whereby the resulting  
25 skins have surface structures resembling those of leather (e.g.,  
grain). The molds further preferably have convex and/or concave  
surface profiles, so that the skins formed therein have surface  
profiles of the type customary with automotive interior parts,  
such as dashboards, door linings or column linings.

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The surfaces of the molds to which the aqueous dispersion of  
polyurethane (A) is applied are free of any coating with a salt  
or electrolyte which would cause the aqueous dispersion of  
polyurethane (A) to coagulate.

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In general, there is no need for a specific pretreatment of the  
surfaces to remove traces of adhering electrolyte quantities,  
since traces of electrolytes are not sufficient to coagulate  
polyurethane dispersions. It suffices for example to clean the  
40 surfaces of the molds prior to their first use in the way which  
is commonly customary with the generally known reaction injection  
molding (RIM) process employing a conventional 2-component  
polyurethane system. For example, the surface of the mold is  
45 washed prior to its 1st use with an aqueous solution which  
contains a customary detergent, rinsed off with tap water and  
dried. After the first skin has been produced by the process of  
the invention and removed from the mold, there is generally no

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need for any cleaning before the mold is recoated with the polyurethane dispersion to produce the next skin.

- 5 To facilitate the removal of the finished skin from the mold, the mold surface is wetted with a commercially available demolding agent, for example a dispersion of waxes in base oils with and without silicone additives, prior to the coating with the polyurethane dispersion.
- 10 Any known process is suitable for applying the polyurethane dispersion, for example dipping. Preferably, the aqueous dispersion of polyurethane (A) is sprayed onto the mold. Suitable for this purpose are the generally known spray guns used for example in the application of paints.
- 15

- Step (I) can be carried out particularly efficiently by heating the aqueous dispersion of polyurethane (A) directly before the dispersion is spray dispensed, for example by spray dispensing the dispersion with pressurized hot steam. Suitable apparatus is described in EP-A-0291850, for example. This method makes it possible to shorten the time required for drying the dispersion film applied to the mold in this way.
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- 25 The dispersion film produced in step (I) is preferably dried by heating the mold surface to 20-150°C, preferably 50-80°C. At these temperatures, drying times from 0.5 to 5 min are sufficient. Drying is generally complete when the skin layer formed has a water content of less than 1% by weight.
- 30

- The sheetlike application of the aqueous dispersion of polyurethane (A) is customarily effected in such amounts that a single performance of steps (I) and (II) will produce a single-layered skin from 10  $\mu$ m to 2 mm in thickness.
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- If thicker skins are desired especially, the first coating and drying step (steps I and II) can be followed by further ones to produce a skin built up from a plurality of skin layers.
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- This is accomplished by

- III. producing a skin composed of a plurality of skin layers by producing on the mold, obtained in step II, coated with a single-layer skin a further sheetlike layer of an aqueous dispersion comprising a polyurethane (A), and
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IV. drying the new film, formed in step (III), of the aqueous dispersion comprising a polyurethane (A) to form a further skin layer, and

- 5 v. optionally repeating steps (III) and (IV) alternately until the desired skin thickness has been reached.

10 It is generally sufficient for steps (III) and (IV) to be repeated from 5 to 50, preferably from 10 to 50, times in order that a skin about 1 mm in thickness may be produced in a particularly efficient way.

15 As to how said steps (III) and (IV) are carried out, the remarks made in relation to steps (I) and (II) apply mutatis mutandis.

The skins preferably have a surface hardness from Shore A 55 to Shore D 80, determined in accordance with German standard specification DIN 5305.

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Particularly suitable aqueous dispersions for producing the skins comprise a polyurethane (A) polymerized from

- 25 a) diisocyanates having from 4 to 30 carbon atoms,

b) diols of which

- 30 b1) from 10 to 100 mol%, based on total diols (b), have a molecular weight from 500 to 5000, and

b2) from 0 to 90 mol%, based on total diols (b), have a molecular weight from 60 to 500 g/mol,

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- c) monomers, other than monomers (a) and (b), which contain at least one isocyanate group or at least one isocyanate reactive group and which in addition contain at least one hydrophilic group or a potentially hydrophilic group to render the polyurethanes water-dispersible,

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- d) optionally further polyfunctional compounds, other than monomers (a) to (c), having reactive groups comprising alcoholic hydroxyl groups, primary or secondary amino groups or isocyanate groups, and

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- e) optionally monofunctional compounds, other than monomers (a) to (d), having a reactive group comprising an alcoholic hydroxyl group, a primary or secondary amino group or an isocyanate group.

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Suitable monomers (a) include the aliphatic or aromatic diisocyanates customarily used in polyurethane chemistry. Preference is given to the monomers (a), or mixtures thereof, which are mentioned as monomers (a) in DE-A-19521500, too.

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Monomers (b) and (d) are preferably the monomers (b) and (d) mentioned in DE-A-19521500.

- 15 Monomers b1 include for example polyesterdiols or polyetherdiols.

Monomers b2 are for example aliphatic diols having from 2 to 12 carbon atoms, e.g., 1,4-butanediol or 1,6-hexanediol.

- 20 Monomers (d) are for example aliphatic amines having from 2 to 12 carbon atoms and from 2 to 4 groups selected from primary or secondary amino groups. Examples are ethylenediamine, isophoronediamine and diethylenetriamine.

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- To ensure that the polyurethanes are water-dispersible, the polyurethanes are polymerized not only from the components (a), (b) and (d) but also from monomers (c) which differ from said components (a), (b) and (d) and which contain at least one isocyanate group or at least one isocyanate reactive group and in addition at least one hydrophilic group or a group which is convertible into hydrophilic groups. In what follows, the expression "hydrophilic groups or potentially hydrophilic groups" is abbreviated to "(potentially) hydrophilic groups". The (potentially) hydrophilic groups react significantly slower with isocyanates than the functional groups of the monomers which are used for forming the polymer main chain.

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- Preferred monomers (c) are again the monomers (c) identified in DE-A-19521500.

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- The proportion of components (a), (b), (c), (d) and (e) accounted for by components having (potentially) hydrophilic groups is generally determined in such a way that the molar quantity of the (potentially) hydrophilic groups is from 80 to 1200, preferably

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140 to 1000, particularly preferably from 200 to 800, mmol/kg, based on the weight quantity of all monomers (a) to (e).

- 5 The (potentially) hydrophilic groups can be nonionic groups, for example polyethylene oxide groups, or preferably (potentially) ionic hydrophilic groups, for example carboxylate or sulfonate groups. Preference is given to operating without effective amounts of nonionic groups.
- 10 The level of nonionic hydrophilic groups, if such groups are incorporated, is generally up to 5, preferably up to 3, particularly preferably up to 1, % by weight, based on the weight quantity of all monomers (a) to (e).
- 15 Monomers (e), the use of which is optional, are monoisocyanates, monoalcohols and monoprimary and secondary amines. In general, their proportion does not exceed 10 mol%, based on the total molar quantity of the monomers. These monofunctional compounds
- 20 customarily bear further functional groups such as carbonyl groups and are used for incorporating into the polyurethane functional groups which render the dispersion or crosslinking or further polymer-analogous reaction of the polyurethane possible.
- 25 It is common knowledge in the field of polyurethane chemistry how the molecular weight of the polyurethanes can be adjusted through choice of the proportions of mutually reactive monomers and the arithmetic mean of the number of reactive functional groups per molecule.
- 30 The components (a) to (e) and their respective molar quantities are normally chosen so that the ratio A : B where
- 35 A) is the molar quantity of isocyanate groups, and
- B) is the sum total of the molar quantity of the hydroxyl groups and the molar quantity of the functional groups capable of reacting with isocyanates in an addition reaction
- 40 is within the range from 0.5 : 1 to 2 : 1, preferably within the range from 0.8 : 1 to 1.5, particularly preferably within the range from 0.9 : 1 to 1.2 : 1. The A : B ratio is most preferably very close to 1 : 1.
- 45

Furthermore, the proportion of monomers (a) is preferably chosen so that the proportion of monomers (a) to (e) which is accounted for by monomers (a) is within the range from 20 to 70% by weight.

- 5 The monomers (a) to (e) used on average bear customarily from 1.5 to 2.5, preferably from 1.9 to 2.1, particularly preferably 2.0, isocyanate groups or functional groups capable of reacting with isocyanates in an addition reaction.
- 10 The polyaddition of components (a) to (e) is generally effected at reaction temperatures from 20 to 180°C, preferably from 50 to 150°C, under atmospheric pressure or under autogenous pressure.
- 15 The reaction times required can range from a few minutes to several hours. In the field of polyurethane chemistry it is known how the reaction time is influenced by a multiplicity of parameters such as temperature, concentration of the monomers, reactivity of the monomers.
- 20 To speed up the reaction of the diisocyanates it is possible to use the customary catalysts, such as dibutyltin dilaurate, tin(II) octoate or diazabicyclo(2.2.2)octane.
- 25 Suitable polymerization apparatus includes stirred tanks or the otherwise customary polymerization apparatus.

- Preferred solvents are miscible with water in any proportion, have an atmospheric-pressure boiling point within the range from 40 to 100°C, and react only slowly with the monomers, if at all.

The dispersions are usually prepared according to one of the following processes:

- 35 The "acetone process" comprises preparing an ionic polyurethane from the components (a) to (c) in a water-miscible solvent which has an atmospheric-pressure boiling point below 100°C. Sufficient water is added to form a dispersion in which water is the coherent phase.
- 40 The "prepolymer mixing process" differs from the acetone process in that initially a prepolymer bearing isocyanate groups is produced and not a fully reacted (potentially) ionic polyurethane. The components here are chosen so that the defined
- 45 ratio of A:B is within the range from greater than 1.0 to 3, preferably within the range from 1.05 to 1.5. The prepolymer is first dispersed in water and then optionally crosslinked or

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branched by reaction of the isocyanate groups with amines bearing more than 2 isocyanate reactive amino groups or extended by reaction with amines bearing 2 isocyanate reactive amino groups. Chain extension takes place even when no amine is added. In this case, isocyanate groups are hydrolyzed to amine groups which react with any remaining isocyanate groups on the prepolymers, resulting in chain extension.

10 Customarily, if a solvent was used in the production of the polyurethane, the bulk of the solvent is removed from the dispersion, for example by distillation under reduced pressure. The dispersions preferably have a solvent content of less than 10% by weight and are particularly preferably free from solvents.

15 Particularly preferred dispersions are described for example in DE-A-2645779 and DE-A-2651506 and also EP-A-0300335. Preferred commercial products are Astacin® Finish PUD or PUM from BASF Aktiengesellschaft.

20 The dispersions preferably contain no substances with which the dispersions are customarily modified into heat coagulable dispersions, i.e., substances as described in EP-A-0257225. The dispersions used in the process of the invention thus preferably  
25 contain no effective amounts of water-soluble polyether polyurethanes as described for example in DE-A 2516979, 2534304 and 3330197 or the electrolytes (metal salts) mentioned in EP-A-0257225.

30 The dispersions of the invention may contain commercially available auxiliaries and additives such as wetting agents, defoamers, delustrants, emulsifiers, thickeners and thixotropiziers, colorants such as dyes and pigments.

35 The skins of the invention are customarily backfoamed. Backfoaming can take place in the mold in which the skins were produced or the skins are removed from this mold and placed for this operation into another mold which is suitable for the expansion of polyurethane.  
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Customarily, the polyurethane foam (B) is applied to that side of the skin which was not in contact with the mold.

45 The production of foams is common knowledge.

The polyurethane foam (B) is advantageously applied by spraying or casting a polyurethane reaction mixture comprising a blowing agent onto the skin surface.

- 5 The polyurethane foam (B) generally is from 1 to 10, preferably from 1 to 7, particularly preferably from 2 to 3, mm in thickness. The polyurethane foam (B) is preferably an open-celled semirigid foam.
- 10 The isocyanate component is for example an isocyanate component also useful as monomer (a) for the production of the aqueous dispersion comprising a polyurethane (A). Particular preference is given to polymeric MDI (methylene diphenyl isocyanate) (e.g.,
- 15 Lupranat® M20 from BASF Aktiengesellschaft).

The polyol component is likewise selected from those which are useful as monomers (a) for the production of the aqueous dispersion comprising a polyurethane (A).

- 20 The blowing agent can be water.

- Particular preference is given to water-blown polyether systems from 50 to 180 kg/m<sup>3</sup> in density. A particularly suitable system
- 25 for producing the polyurethane foam is a commercially available system for producing semirigid foams such as, for example the Elastoflex® E systems (commercial product from Elastogran GmbH, Lemförde).

- 30 Molds and application techniques suitable for the backfoaming of the skins are described for example in WO 93/23237 and JP A-S63-256409.

- 35 Optionally, the skins, especially when the polyurethane (A) is predominantly derived from aromatic isocyanates as monomers (a), are coated with an IMP coating for stabilization against UV radiation or with another customary coating. IMP coatings are commercially available 2-component systems. Component 1 contains
- 40 for example a solution of a polyurethane resin which, as well as alcohol, contains customary organic solvents such as, for example, aromatic hydrocarbons and esters (e.g., xylenes, n-butyl acetate or 1-methoxy-2-propyl acetate). Component 2 (curing agent component) can be an aliphatic isocyanate, for example. IMP
- 45 coatings are available for example from ISL-Chemie GmbH, Kürten,

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as a 2-component system under the tradenames of Isothan-TMP-2K coating and Isothan curing agent.

- 5 The skins produced in this way are especially useful as automotive interior parts, such as dashboards, door linings or column linings.

#### Experimental part

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#### Example A:

- First the open mold was prepared by using a heating unit to raise the mold surface to a temperature of  $75 \pm 5^\circ\text{C}$ . The instrument panel surface to be sprayed was then provided with a wax dispersion in a base oil as demolding agent, Acmosil® 56-4566 (from Acmos Chemie, Bremen) or Bomix® 6389/8 (from Böttler, Telgte).
- 20 Astacin Finish PUM from BASF AG, a polyurethane dispersion comprising an anionic-functional polyurethane derived from polyesterdiol and aliphatic isocyanates, was admixed with 10% of color, BASF Lepton Blue, and introduced into a spray gun pressure vessel. The sprayer of the type HS-25 is from Krautzberger GmbH,
- 25 Eltville a.R., with a mini extension  $90^\circ\text{C}$  - for VD nozzles - it was used to spray the undercuts repeatedly at low settings, about 10-20 mμ per spraying operation. The larger areas were sprayed using a sprayer of the type Perfekt HS-25, A 10, at a higher setting, about 30 - 50 mμ per spraying operation. These two
- 30 spraying operations were repeated until a skin of about  $0.9 \pm 0.3$  mm had been formed. Each spraying operation was followed by an interval, during which all the water evaporated, until the next spraying operation was carried out. The finished skin was conditioned for 5 min by surface heat (conditioning oven), then
- 35 demolded and appropriately stored for further processing.

The properties of the skins produced in this way are reported in Table 1.

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#### Example B:

- 45 The PU skin was placed into an expansion mold with lid and pulled flat against the wall by means of a vacuum. The lid was provided with a wax-based demolding agent (Acmos), and thereafter an insert of plastic or metal was attached in such a way that the

later closed-lid foaming operation would cause the foam to form a unit with skin and insert.

The open mold was entered, with the aid of a Puromat -  
 5 two-component machine - from Elastogran Dosiertechnik, with an  
 energy-absorbing, semirigid foam of the type Elastoflex® E 3320.  
 Elastoflex® E is a two-component water-blown MDI system from the  
 automotive and specialties division of Elastogran GmbH (foam  
 properties see Table 2). The closed lid caused the foam in the  
 10 mold to be compressed to a wall thickness of 2 cm, and after  
 3 min the foam was removed as a complete structural component  
 from the opened mold (foam properties see Table 2).

15 Table 1

Typical properties of sprayed skin:

20			Skin A1	Skin A2
	Density	kg/m <sup>3</sup>	1010	990
	Tensile strength	kPa	14440	11360
	Elongation	%	780	710
	Tear strength	N/mm	37	36
25	Fogging	mg	2.2	1.5
	Heat test	120°C	O.K.	O.K.

30 Table 2

Typical properties of foam B:

35	Density	kg/m <sup>3</sup>	110
	Tensile strength	kPa	320
	Elongation	%	45
40	Compression hardness	kPa	
	at 20%		38
	at 40%		61
	at 60%		120

We claim:

- 5 1. A process for producing skins from polyurethane dispersions, which comprises
  - 10 I. producing on a mold a sheetlike layer of an aqueous dispersion comprising a polyurethane (A), the mold surface being free of any electrolyte which would cause the aqueous dispersion comprising a polyurethane (A) to coagulate,
  - 15 II. drying the film, produced in step (I), of the aqueous dispersion comprising a polyurethane (A) to obtain a single-layered skin,
  - 20 III. optionally producing a skin composed of a plurality of skin layers by producing on the mold, obtained in step II, coated with a single-layered skin a further sheetlike layer of an aqueous dispersion comprising a polyurethane (A),
  - 25 IV. drying the new film, formed in step (III), of the aqueous dispersion comprising a polyurethane (A) to form a further skin layer, and
  - 30 V. optionally repeating steps (III) and (IV) from 5 to 50 times.
- 35 2. A process as claimed in claim 1, further comprising a step (VI) of applying a layer of a polyurethane foam (B) to the skin on that side which was not in contact with the surface of the mold.
- 40 3. A process as claimed in claim 1 or 2, wherein the skin is placed into another, sealable mold and a support is backfoamed with the polyurethane foam (B).
- 45 4. A process as claimed in any of claims 1 to 3, wherein the amount of aqueous dispersion comprising a polyurethane (A) applied per coating operation (step I or III) is such that the respective skin layer has a thickness from 10  $\mu$ m to 2 mm.

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5. A process as claimed in any of claims 1 to 4, wherein skins from 10  $\mu$ m to 4 mm in thickness are produced.
- 5 6. A process as claimed in any of claims 1 to 5, wherein the aqueous dispersion comprising a polyurethane (A) is applied in steps (I) and (III) by spraying.
- 10 7. A process as claimed in any of claims 2 to 6, wherein the layer of polyurethane foam (B) is from 1 to 7 mm in thickness.
- 15 8. A process as claimed in any of claims 1 to 7, wherein the polyurethane foam (B) is applied to the surface of the skin by spraying a polyurethane reaction mixture comprising a blowing agent onto the skin surface.
- 20 9. A process as claimed in any of claims 1 to 8, wherein the polyurethane (A) in the aqueous dispersions comprising a polyurethane (A) is polymerized from
- a) diisocyanates having from 4 to 30 carbon atoms,
- 25 b) diols of which
- b1) from 10 to 100 mol%, based on total diols (b), have a molecular weight from 500 to 5000, and
- 30 b2) from 0 to 90 mol%, based on total diols (b), have a molecular weight from 60 to 500 g/mol,
- 35 c) monomers, other than monomers (a) and (b), which contain at least one isocyanate group or at least one isocyanate reactive group and which in addition contain at least one hydrophilic group or a potentially hydrophilic group to render the polyurethanes water-dispersible,
- 40 d) optionally further polyfunctional compounds, other than monomers (a) to (c), having reactive groups comprising alcoholic hydroxyl groups, primary or secondary amino groups or isocyanate groups, and



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- e) optionally monofunctional compounds, other than monomers (a) to (d), having a reactive group comprising an alcoholic hydroxyl group, a primary or secondary amino group or an isocyanate group.

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10. A process as claimed in any of claims 2 to 9, wherein the polyurethane foam is produced using a commercially available system for producing semirigid foams.

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